IS 5281 (1979): Fenitrothion Emulsifiable Concentrates [FAD 1: Pesticides and Pesticides Residue Analysis]

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(Reaffirmed - 2012)

Indian Standard

SPECIFICATION FOR FENITROTHION EMULSIFIABLE CONCENTRATES

(First Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1980

Indian Standard

SPECIFICATION FOR FENITROTHION EMULSIFIABLE CONCENTRATES

(First Revision)

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AMENDMENT NO. 1 MAY 1994
TO
IS 5281 : 1979 SPECIFICATION FOR FENITROTHION EC

(First Revision)

(*Page* 6, *clause* **4.1**) -- Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **2.3.1** of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR FENITROTHION EMULSIFIABLE CONCENTRATES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 October 1979, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Fenitrothion emulsifiable concentrates are largely used in the control of insect pests of agricultural and public health importance.

0.3 Fenitrothion emulsifiable concentrate formulations are generally manufactured to contain 50 percent (*m/m*) and 82·5 percent (*m/m*) of fenitrothion. 82·5 percent (*m/m*), emulsifiable concentrate is used for aerial spraying.

0.4 This standard was first published in 1969. Six amendments to this were issued from time to time. This revision incorporates six amendments issued to the earlier version and test method has been modified.

0.5 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for fenitrothion emulsifiable concentrates.

2. REQUIREMENTS

2.1 Constituents — The material shall consist of fenitrothion, technical, dissolved in suitable solvent(s), together with emulsifying agent(s), and with or without stabilizer(s).

2.1.1 Fenitrothion, technical, employed in the manufacture of emulsifiable concentrates shall conform to IS : 5280-1969*.

2.2 Physical — The material shall comply with the physical requirements specified in **2.2.1** to **2.2.4**.

2.2.1 *Description* — The material shall be in the form of clear, stable homogeneous liquid, free from foreign matter. Suspended matter shall be negligible. It shall form a suspension suitable for spray on dilution with water.

2.2.2 *Cold Test* — No turbidity or separation of solid or oily matter or both shall occur when the material is subjected to the cold test at 10°C as prescribed in **13.1** of IS : 6940-1973†, or at any other lower temperature as agreed to between the purchaser and the supplier. Introduction of a seeding crystal is not necessary for the test.

2.2.3 *Flash Point (Abel)* — When determined by the method prescribed in IS : 1448 [P : 20]-1960‡, the flash point of the material shall be above 24·5°C.

2.2.4 *Emulsion Stability* — Any separation, including creaming at the top and sedimentation at the bottom of 100 ml of emulsion prepared in standard hard water with 5 ml of the concentrate for public health use and with 2 ml of concentrate for agricultural use, shall not exceed 2 ml when tested by one of the methods prescribed in **13.3** of IS : 6940-1973†.

2.3 Chemical — The material shall comply with the chemical requirements specified in **2.3.1** and **2.3.2**.

*Specification for fenitrothion, technical.

†Methods of tests for pesticides and their formulations.

‡Methods of tests for petroleum and its products, P : 20 Flash point by Able apparatus.

2.3.1 *Fenitrothion Content* — When determined by the method prescribed in Appendix A, the observed fenitrothion content, percent (*m/m*), of any of the samples shall not differ from the declared nominal value by more than the percent tolerance limits indicated below:

Nominal Value, Percent	*Tolerance*	
Up to 9	+10 − 5	percent of the nominal value
10 and below 50	± 5	
50 and above	+ 5 − 3	

2.3.1.1 The actual value of fenitrothion content in the formulation shall be calculated to the second decimal place for rounding off to the first decimal place before applying the tolerances given in **2.3.1.**

2.3.2 *Acidity or Alkalinity* — When tested by the method prescribed in **11.3** of IS : 6940-1973*, acidity (as H_2SO_4), or alkalinity (as NaOH) of the material shall be not more than 1·0 and 0·5 percent by mass respectively.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part II)-1976†.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other information as is necessary under the Insecticides Act and Rules:

a) Name of the material and its end use;

b) Name of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Net mass of contents;

f) Nominal fenitrothion content, percent (*m/m*); and

g) The minimum cautionary notice worded as in the Insecticides Act and Rules.

*Methods of tests for pesticides and their formulations.

†Requirements for packing of pesticides: Part II Liquid pesticides.

3.2.1 Each container may also be marked with the ISI certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard Methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.2.1** to **2.2.4**, **2.3.1** and **2.3.2**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 2.3.1)

DETERMINATION OF FENITROTHION CONTENT

A-0. PRINCIPLE

A-0.1 Fenitrothion is separated from the most likely impurity *p*-nitro-*m*-cresol by treating the benzene solution with chilled (10°C) 10 percent sodium carbonate solution. The benzene solution of fenitrothion is reduced by zinc and acetic acid-hydrochloric acid mixture. The amine groups formed are titrated with standard sodium nitrite solution.

*Specification for water for general laboratory use (*second revision*).

A-1. REAGENTS

A-1.1 Benzene

A-1.2 Sodium Carbonate Solution — 10 percent, chilled (10°C).

A-1.3 Sodium Sulphate — anhydrous.

A-1.4 Acetic Acid-Hydrochloric Acid Mixture — prepared by mixing glacial acetic acid and concentrated hydrochloric acid (9 : 1 *v/v*).

A-1.5 Zinc Dust — iron free.

A-1.6 Acetic Acid — glacial.

A-1.7 Hydrochloric Acid — concentrated.

A-1.8 Sodium or Potassium Bromide

A-1.9 Sulphanilic Acid

A-1.10 Standard Sodium Nitrite Solution — 0·1 M. Prepared by dissolving 6·90 g of sodium nitrite in water and making up to one litre with water. Standardize as follows:

Weigh accurately 0·40 to 0·45 g of anhydrous sulphanilic acid into a 400-ml tall-form beaker. Add 80 ml of distilled water, 10 ml of concentrated hydrochloric acid, 30 ml of glacial acetic acid and 5 g of sodium (or potassium) bromide. Cool the mixture to 0 to 10°C by the addition of clean, shaved ice and stir mechanically. Titrate at 0 to 10°C with the 0·1 M sodium nitrite as rapidly as the spot test permits. Near the end-point, add the sodium nitrite in four-drop portions.

$$\text{Normality of 0·1 M sodium nitrite} = \frac{a}{b} - 5{\cdot}774$$

where

a = mass in g of sulphanilic acid used, and

b = volume in ml of 0·1 M sodium nitrite required.

A-2. PROCEDURE

A-2.1 Separation and Extractions of Fenitrothion — To a 250 ml separatory funnel containing 100-ml benzene and 1 to 2 g of anhydrous sodium sulphate, add accurately weighed sample containing 0·8 to 1·0 g of fenitrothion. Add 25 ml of 10 percent chilled sodium carbonate solution and very *carefully and slowly* extract the free *p*-nitro-*m*-cresol by swirling or mixing the contents very slowly to avoid emulsion formation.

Vigorous shaking shall be avoided. Drain out the clear yellow coloured aqueous layer. If any emulsion formation is observed then emulsion shall not be discarded but retained with the solvent phase. Add again 1 to 2 g of anhydrous sodium sulphate followed by chilled 10 percent sodium carbonate solution and the extraction of the free *p*-nitro-*m*-cresol is carried out as described above. Continue the extraction with sodium carbonate solution till all the *p*-nitro-*m*-cresol is extracted as indicated by absence of yellow colour to the aqueous phase.

A-2.2 Determination of Fenitrothion Content — Transfer the benzene layer to 500-ml round bottom flask quantitatively by washing the saparatory funnel 4 times with 25 ml portions of benzene. Remove the solvent by distillatin. Add 100 ml of acetic acid-hydrochloric acid mixture and 6 g of iron free zinc dust. Cover the flask with a funnel and heat on a steam bath for 30 minutes. Add 50 ml of concentrated hydrochloric acid and continue the heating till the zinc is completely dissolved. Cool and transfer the contents of round bottom flask into a tall-form beaker quantitatively by washing with distilled water. Add 5 g of sodium or potassium bromide. Cool to 0° to 10°C by the addition of clean shaved ice and place under mechanical stirring. Titrate the mixture at 0° to 10°C with the standardized 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end-point add the sodium nitrite solution in four-drop portions.

A-2.3 Spot Test — Dip a glass rod into the solution to be tested and then touch the rod quickly to a piece of potassium iodide-starch paper. The end-point is reached when the intense blue-black colour appears within 15 to 20 seconds and can be obtained repeatedly during a one-minute period without further addition of sodium nitrite solution.

A-3. CALCULATION

A-3.1 Fenitrothion content, percent by mass $= \frac{VN \times 27{\cdot}72 \times f}{M}$

where

V = volume (in ml) of 0·1 M sodium nitrite solution required for the test;

N = normality of 0·1 M sodium nitrite solution;

f = correction factor T/A:

T = theoretical calculated quantity of 0·1 M sodium nitrite solution to be used in the nitro-group determination of 1·00 g of a pure and solid substance containing one nitro group; and

A = actual quantity of 0·1 M sodium nitrite solution used in the nitro-group determination of 1·00 g of pure, recrystallized sample of the same substance, following the above described method and using the same reagents.

M = mass (in g) of the material taken for the test.

NOTE — The correction factor f includes the errors arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall lie within the range of 0·98 to 1·02.

INDIAN STANDARDS

ON

PESTICIDES (EMULSIFIABLE CONCENTRATES)

IS:

632-1978 Gamma-BHC (HCH) emulsifiable concentrates (*fourth revision*)
633-1975 DDT emulsifiable concentrates (*first revision*)
1307-1973 Aldrin emulsifiable concentrates (*first revision*)
1310-1974 Endrin emulsifiable concentrates (*first revision*)
2567-1978 Malathion emulsifiable concentrates (*second revision*)
2682-1966 Chlordane emulsifiable concentrates (*first revision*)
2861-1964 Diazinon emulsifiable concentrates
2965-1978 Methyl parathion emulsifiable concentrates (*first revision*)
3903-1975 Dimethoate emulsifiable concentrates (*first revision*)
3905-1966 Thiometon emulsifiable concentrates
4323-1967 Endosulfan emulsifiable concentrates
4325-1967 Binapacryl emulsifiable concentrates
4808-1968 Pyrethrum emulsifiable concentrates
5277-1978 Dichlorvos emulsifiable concentrates (*first revision*)
5279-1969 Dicofol emulsifiable concentrates
5281-1969 Fenitrothion emulsifiable concentrates
6439-1978 Heptachlor emulsifiable concentrates (*first revision*)
7946-1976 Toxaphene emulsifiable concentrates
6177-1971 Phosphamidon water soluble concentrates
7948-1976 Fenthion emulsifiable concentrates
8026-1976 Formothion emulsifiable concentrates
8027-1976 Propanil emulsifiable concentrates
8028-1976 Quinalphos emulsifiable concentrates
8074-1976 Monocrotophos water soluble concentrates
8259-1976 Oxydemeton-methyl emulsifiable concentrates
8497-1977 Paraquat dichloride salt aqueous solutions
8291-1976 Phenthoate emulsifiable concentrates
8487-1977 Phosalone emulsifiable concentrates
8498-1977 Temephos emulsifiable concentrates

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure stress	pascal	Pa	1 Pa = 1 N/m^2

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